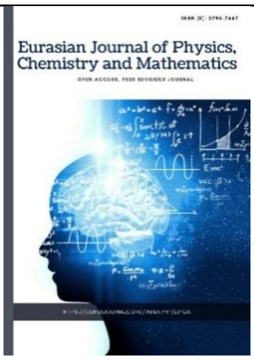

Synthesis Of Biodegradable Hydrogels From Polyacrylonitrile (Pan)-Based Carpet Waste And Cotton Linter And Their Application In Agriculture

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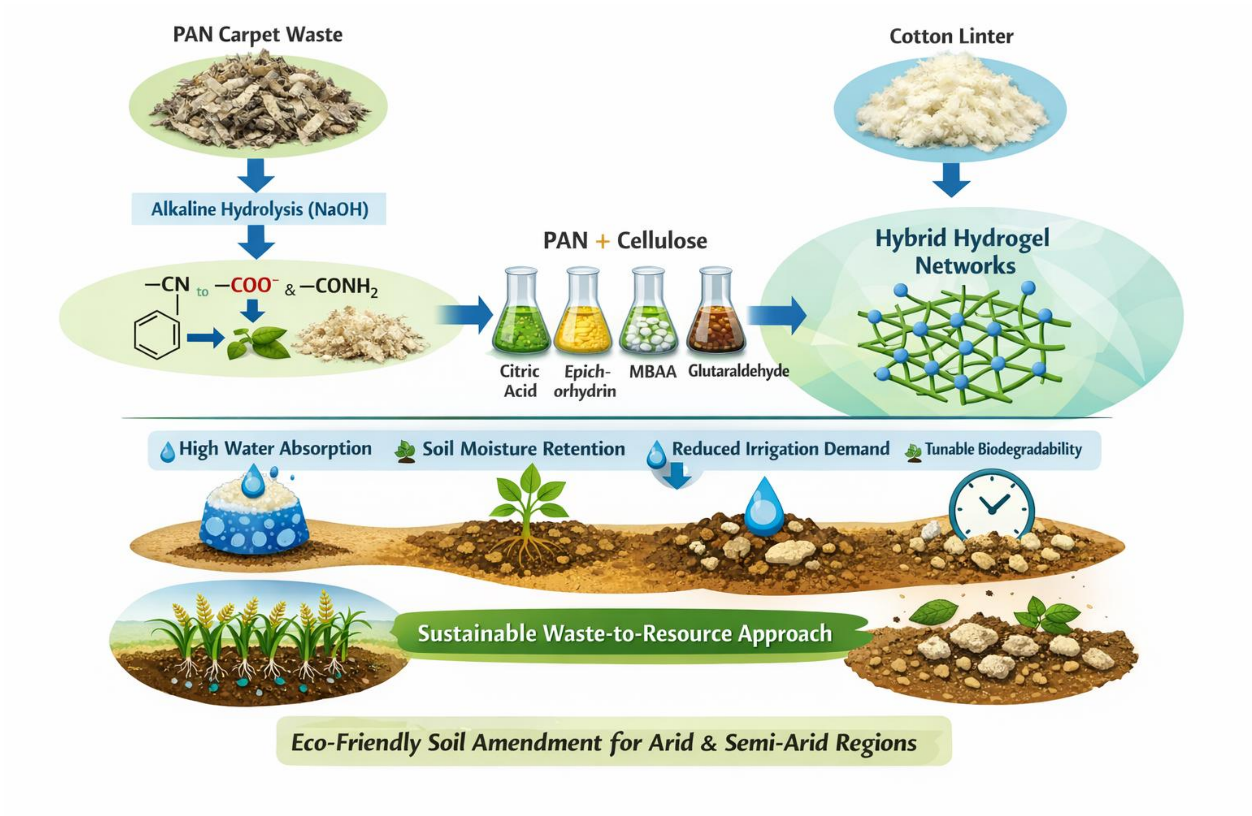
ABSTRACT

Graphical Abstract Schematic illustration of a sustainable waste-to-resource approach for the synthesis of hybrid biodegradable hydrogels from PAN-based carpet manufacturing waste and cotton linter. Alkaline hydrolysis of PAN introduces hydrophilic carboxylate and amide groups, followed by crosslinking with different agents (citric acid, epichlorohydrin, MBAA, and glutaraldehyde) to form three-dimensional PAN–cellulose networks. The resulting hydrogels exhibit high water absorption, improved soil moisture retention, reduced irrigation demand, and tunable biodegradability, demonstrating their potential as eco-friendly soil amendments for sustainable agriculture in arid and semi-arid regions.

Hybrid biodegradable hydrogels were synthesized from polyacrylonitrile (PAN)-based carpet manufacturing waste and cotton linter for sustainable agricultural water management. PAN waste was alkaline-hydrolyzed to introduce hydrophilic carboxylate and amide groups and subsequently combined with cellulose-rich cotton linter. The resulting PAN–cellulose systems were crosslinked using different agents, including N,N′-methylenebisacrylamide, epichlorohydrin, citric acid, and glutaraldehyde, to tailor network structure and performance. The hydrogels exhibited rapid swelling and diffusion-controlled kinetics, with equilibrium swelling strongly dependent on crosslinker type. Citric-acid- and epichlorohydrin-crosslinked hydrogels showed the highest transient swelling capacities (up to ≈740 g/g) and high equilibrium swelling under stabilized conditions, superior soil moisture retention, and favorable biodegradability. Soil application of these hydrogels reduced water loss by approximately 25–30% compared to untreated soil. In contrast, glutaraldehyde-crosslinked hydrogels displayed limited swelling and biodegradability due to their rigid network structure. The results demonstrate that PAN–cellulose hydrogels derived from carpet waste represent an effective waste-to-resource strategy and a promising eco-friendly soil amendment for water-saving agriculture in arid and semi-arid regions.

MBAA-crosslinked hydrogels exhibited moderate swelling and limited biodegradation, primarily governed by degradation of the cellulose fraction.

Keywords: Hybrid biodegradable



1. Introduction

Uzbekistan is located in a predominantly arid region of Central Asia, characterized by limited precipitation and high rates of evaporation. The country's agricultural sector relies heavily on extensive irrigation networks to maintain crop production due to scarce natural rainfall levels, which average well below 250 mm annually in many regions such as Mirzachoʻl Steppe, where major irrigated cotton and grain production takes place. These climatic conditions exacerbate soil moisture deficits and make sustainable water management critical for agricultural productivity.

Climate change trends in Uzbekistan indicate rising temperatures, increased frequency of droughts, and reduction in available water resources, significantly impacting water availability for agriculture. The effects of climate change are expected to intensify land degradation and desertification, further straining water supplies and agricultural systems. Moreover, future projections suggest that by 2030, Uzbekistan may join the ranks of countries experiencing acute water scarcity, driven by population growth and increasing water demand coupled with decreasing renewable water resources.

Agriculture remains a cornerstone of Uzbekistan's economy, accounting for a significant share of employment and GDP, and depends predominantly on irrigation from transboundary rivers such as the Amu Darya and Syr Darya. However, traditional irrigation practices result in substantial water loss due to inefficient infrastructure and high evaporative losses, and water scarcity poses a direct threat to crop yields and rural livelihoods. Improvements in water conservation technologies and agro-water management strategies are thus critical for sustaining agricultural outputs and enhancing food security under changing climatic conditions in the country.

Hydrogels—three-dimensional crosslinked polymer networks capable of absorbing and retaining large amounts of water—offer a promising solution to enhance soil moisture retention and water use efficiency in arid agricultural systems. Synthetic superabsorbent polymers have been widely studied for this purpose, but their non-biodegradable nature raises environmental concerns. Natural polymer-based hydrogels, derived from renewable sources such as cellulose, present an environmentally compatible alternative due to their biodegradability and non-toxic properties. In particular, cotton linter, a by-product of the cotton processing industry and predominantly composed of cellulose with a repeating unit $(C_6H_{10}O_5)n$, represents an abundant and renewable raw material for sustainable hydrogel synthesis.

Simultaneously, enormous quantities of synthetic textile waste, particularly polyacrylonitrile (PAN)-based carpet manufacturing residues, remain an underutilized resource with environmental disposal challenges. Valorizing PAN waste through chemical modification and integrating it with cellulose from cotton linter into a hybrid hydrogel system can bridge the gap between high performance and biodegradability. This hybrid approach not only provides a pathway for textile and agricultural residue valorization but also addresses critical issues of water scarcity and sustainable agriculture in Uzbekistan’s arid climate.

2. Literature Review

2.1. Hydrogels and Their Role in Agriculture

Hydrogels are three-dimensional crosslinked polymer networks capable of absorbing and retaining large quantities of water due to the presence of hydrophilic functional groups within their structure [1, 2]. The swelling behavior and water retention capacity of hydrogels depend strongly on polymer composition, crosslinking density, and environmental conditions such as pH and ionic strength [2].

In agricultural applications, hydrogels have been widely investigated as soil conditioners that enhance soil water-holding capacity, reduce irrigation frequency, and mitigate drought stress in crops [3, 4]. By acting as micro-reservoirs in the soil, hydrogels improve water use efficiency and contribute to sustainable crop production, particularly in arid and semi-arid regions [3].

However, most commercially available superabsorbent polymers are synthesized from petroleum-based monomers such as acrylic acid and acrylamide, which exhibit limited biodegradability and may persist in soil environments [4, 5]. These limitations have stimulated growing interest in the development of biodegradable and environmentally friendly hydrogel systems derived from renewable or waste-based resources.

2.2. Cellulose-Based Hydrogels and Cotton Linter Utilization

Cellulose is the most abundant natural polymer, composed of repeating anhydroglucose units linked via β-1,4-glycosidic bonds, and is widely recognized for its renewability, biodegradability, and non-toxicity [1, 5]. Due to the presence of multiple hydroxyl (–OH) groups, cellulose and its derivatives exhibit strong hydrophilicity, making them attractive precursors for hydrogel synthesis [6].

Cellulose-based hydrogels have been extensively studied for agricultural applications, where they demonstrate favorable swelling behavior, biodegradability, and compatibility with soil ecosystems [5, 6]. Among various cellulose sources, cotton linter—an agricultural by-product rich in cellulose with a repeating unit of $(C_6H_{10}O_5)n$—has gained attention as an economical and sustainable raw material for hydrogel production [6, 7].

Several studies have reported that hydrogels derived from cotton waste and cellulose-based materials significantly improve soil moisture retention and plant growth while minimizing environmental impact [6, 7]. These findings highlight the potential of cotton linter as a biodegradable component in hybrid hydrogel systems designed for agricultural water management.

2.3. Hybrid Hydrogels from Natural and Synthetic Polymers

Although cellulose-based hydrogels offer excellent environmental compatibility, their mechanical strength and long-term structural stability can be limited when compared to fully synthetic hydrogels [4, 6]. To overcome these limitations, hybrid hydrogel systems combining natural polymers with synthetic polymers have been proposed as an effective strategy to balance performance and biodegradability [6].

Hybrid hydrogels benefit from the mechanical robustness and tunable chemistry of synthetic polymers while retaining the biodegradability and sustainability of natural polymers [3, 6]. Recent research has shown that such systems exhibit improved swelling stability, enhanced mechanical properties, and controlled degradation behavior, making them suitable for agricultural applications [6, 7].

2.4. Valorization of Textile Waste and PAN-Based Materials

The textile industry generates substantial quantities of synthetic polymer waste, particularly polyacrylonitrile (PAN)-based fibers used in carpet manufacturing, which are difficult to degrade and often disposed of by landfilling or incineration [8]. The chemical stability of PAN poses environmental challenges, highlighting the need for innovative recycling and valorization strategies.

Recent studies have demonstrated that chemical modification of PAN, such as alkaline hydrolysis of nitrile (–CN) groups into carboxylate ($–COO^-$) and amide ($–CONH_2$) functionalities, can significantly enhance its hydrophilicity and reactivity [8]. However, the integration of PAN-based textile waste with biodegradable natural polymers such as cellulose for agricultural hydrogel applications remains relatively underexplored, representing a clear research gap.

2.5. Relevance to Water Scarcity and Sustainable Agriculture

Water scarcity is a critical issue in arid regions, including Central Asia and Uzbekistan, where agricultural productivity relies heavily on irrigation [9, 10]. Sustainable water management strategies that improve soil moisture retention are essential for maintaining crop yields under increasing climatic stress.

Hydrogels derived from waste materials provide a promising solution by simultaneously addressing water scarcity and waste management challenges [3, 7, 9]. The development of hybrid hydrogels based on PAN carpet waste and cotton linter aligns with circular economy principles and offers a sustainable approach for enhancing agricultural water use efficiency in water-limited environments [8–10].

3. Materials and Methods

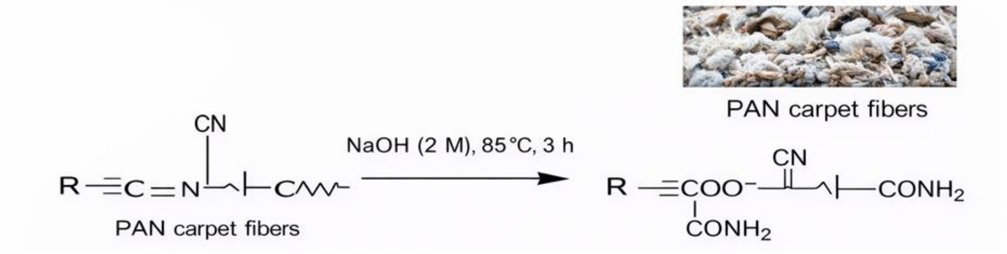
3.1. Materials

Polyacrylonitrile (PAN)-based carpet manufacturing waste (nitron fiber) was collected from local carpet production residues and mechanically shredded to an average fiber length of 3–5 mm. Cotton linter, used as a natural cellulose source, was supplied by a local cotton processing facility and contained more than 95% cellulose with a repeating unit of $(C_6H_{10}O_5)n$.

Sodium hydroxide (NaOH), ammonium persulfate (APS), N,N′-methylenebisacrylamide (MBAA), epichlorohydrin (ECH), citric acid (CA), and glutaraldehyde (GA) were of analytical grade and used without further purification. Distilled water was used throughout all experiments.

3.2. Alkaline Hydrolysis of PAN Carpet Waste

PAN carpet waste was subjected to alkaline hydrolysis to introduce hydrophilic functional groups. Briefly, shredded PAN fibers were dispersed in aqueous NaOH solution (2 M) at a solid-to-liquid ratio of 1:20 (w/v). The mixture was heated at 85 °C under continuous stirring for 3 h.



Scheme 1. Alkaline hydrolysis of PAN carpet waste converting nitrile (–CN) groups into carboxylate ($–COO^-$) and amide ($–CONH_2$) functionalities.

During this process, nitrile (–CN) groups were partially converted into carboxylate ($–COO^-$) and amide ($–CONH_2$) functionalities. After hydrolysis, the modified PAN fibers were repeatedly washed with distilled water until neutral pH was achieved and then dried at 60 °C to constant weight.

3.3. Preparation of Cotton Linter Suspension

Cotton linter was dispersed in distilled water (2 wt%) and mechanically stirred at 80 °C for 1 h to obtain a homogeneous cellulose suspension. This suspension served as the biodegradable component in the hybrid hydrogel system.

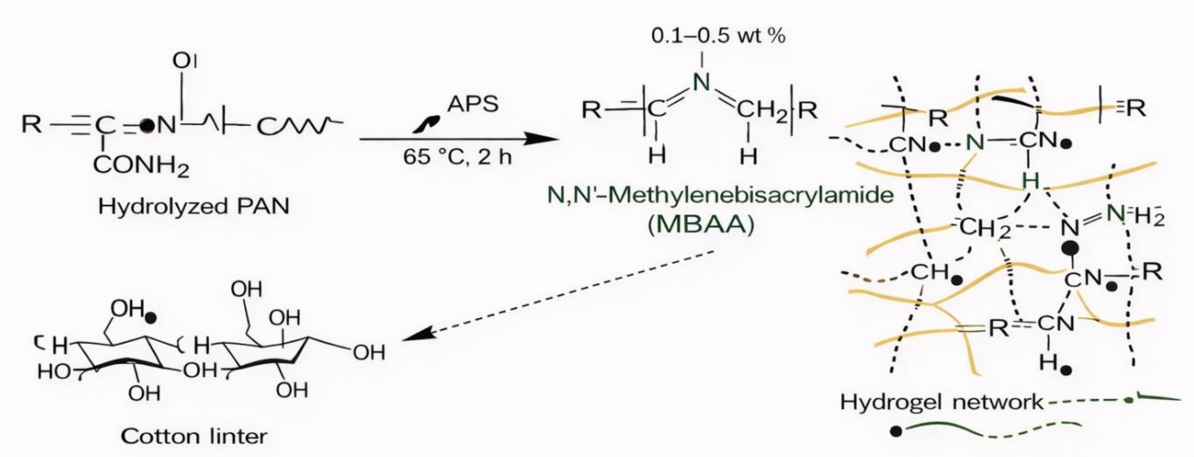
3.4. Synthesis of Hybrid Hydrogels Using Different Crosslinking Agents

Hydrolyzed PAN and cotton linter suspensions were mixed at a PAN:cellulose mass ratio of 70:30 under constant stirring. Free-radical initiation was achieved using ammonium persulfate (APS, 1 wt% relative to total polymer content). Different crosslinking agents were employed to investigate their effect on hydrogel structure and properties.

3.4.1. MBAA-Crosslinked Hydrogels

N,N′-methylenebisacrylamide (MBAA) was added at concentrations of 0.1–0.5 wt% relative to total polymer mass. The reaction mixture was maintained at 65 °C for 2 h to allow covalent crosslinking and formation of a three-dimensional hydrogel network. The resulting hydrogel was washed thoroughly with distilled water to remove unreacted species.

The crosslinking mechanism is schematically shown in Scheme 2.

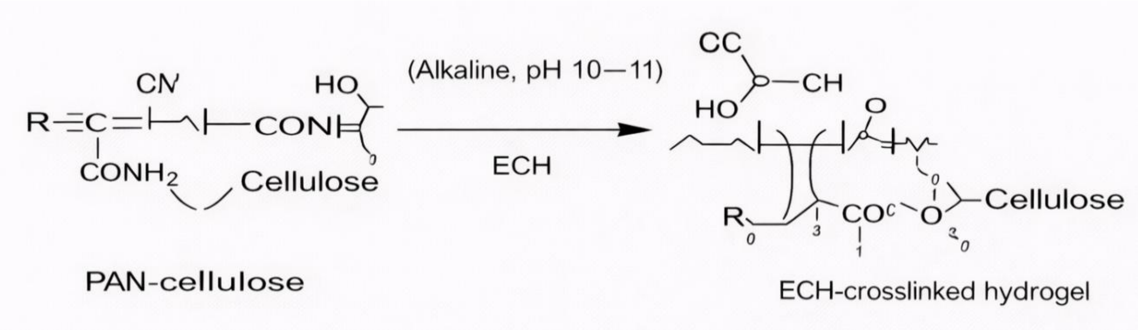


Scheme 2. Free-radical crosslinking of hydrolyzed PAN and cotton linter using MBAA.

3.4.2. Epichlorohydrin (ECH)-Crosslinked Hydrogels

For ECH crosslinking, epichlorohydrin was added dropwise (0.5–2.0 wt%) to the PAN–cellulose mixture under alkaline conditions (pH 10–11). The reaction was carried out at 60 °C for 3 h. ECH primarily reacted with hydroxyl groups of cellulose and carboxylate groups of hydrolyzed PAN, forming ether linkages within the polymer network.

The crosslinking mechanism is schematically shown in Scheme 3.

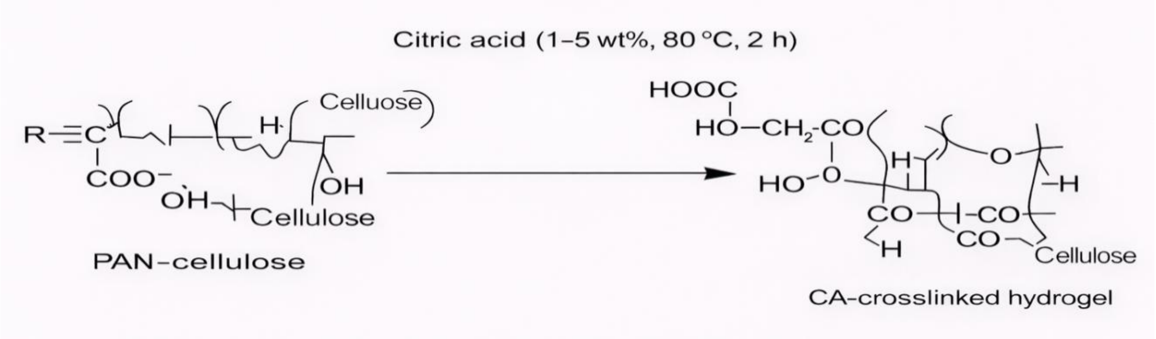


Scheme 3. Epichlorohydrin-mediated ether linkage formation in PAN–cellulose hydrogels.

3.4.3. Citric Acid (CA)-Crosslinked Hydrogels

Citric acid was employed as a bio-based, environmentally friendly crosslinker. Citric acid (1–5 wt%) was added to the PAN–cellulose mixture, followed by heating at 80 °C for 2 h to promote esterification between carboxyl groups of citric acid and hydroxyl groups of cellulose. This method enabled the formation of partially biodegradable hydrogel networks.

The crosslinking mechanism is schematically shown in Scheme 4.

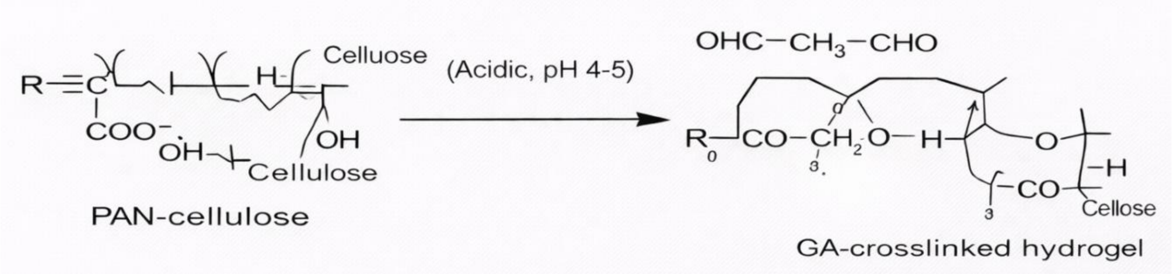


Scheme 4. Citric-acid-based ester crosslinking of PAN–cellulose hydrogels.

3.4.4. Glutaraldehyde (GA)-Crosslinked Hydrogels

Glutaraldehyde was used as an alternative crosslinking agent to enhance network rigidity. GA (0.25–1.0 wt%) was added to the polymer mixture under acidic conditions (pH 4–5), and the reaction was allowed to proceed at 50 °C for 2 h. Crosslinking occurred through the formation of acetal linkages with hydroxyl groups present in cellulose.

The crosslinking mechanism is schematically shown in Scheme 5.



Scheme 5. Glutaraldehyde crosslinking via acetal bond formation.

3.5. Washing and Drying of Hydrogels

All synthesized hydrogels were washed repeatedly with distilled water to remove residual reagents and unreacted crosslinkers. The samples were then dried at 50–60 °C until constant weight and stored in a desiccator prior to characterization.

3.6. Characterization and Performance Evaluation

Water absorption capacity was determined by gravimetric swelling measurements in distilled water at room temperature. Soil moisture retention performance was evaluated by incorporating dried hydrogels into soil samples and monitoring moisture content over time under controlled conditions.

4. Results and Discussion

4.1. Swelling Kinetics and Network Formation

Figure 3 illustrates the swelling kinetics of PAN–cellulose hybrid hydrogels crosslinked with different agents. The presented values correspond to the maximum transient swelling during water uptake, observed within the first 30–90 min before network relaxation and structural stabilization. All samples exhibited diffusion-controlled swelling behavior, followed by gradual stabilization as the polymer networks approached equilibrium.

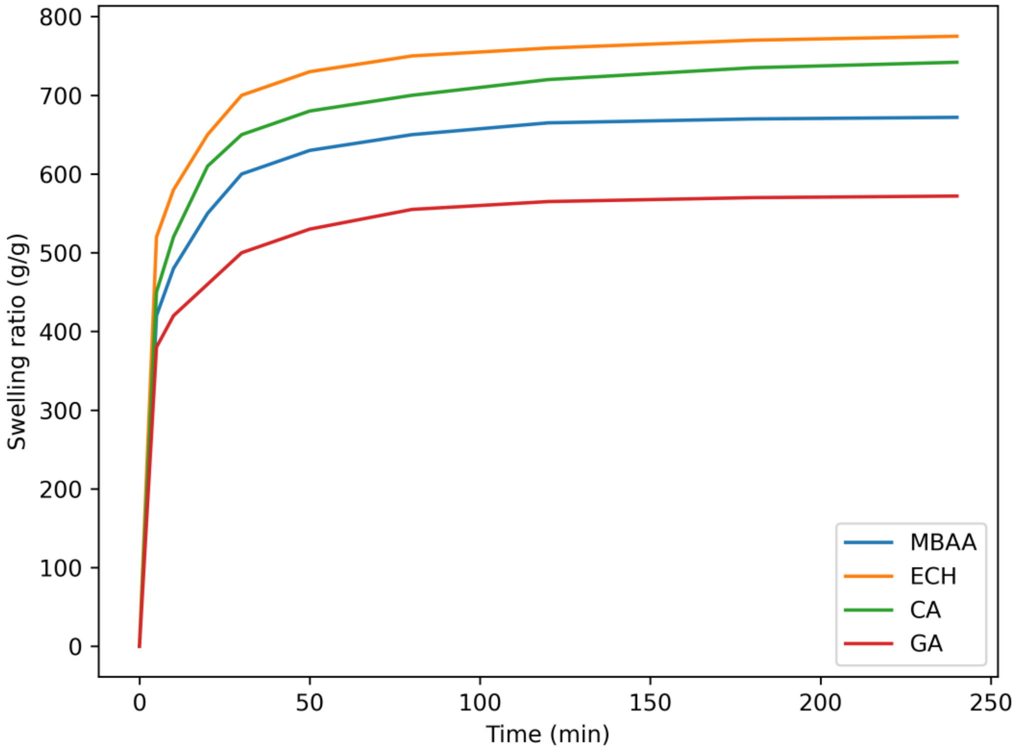


Figure 3. Swelling kinetics of PAN–cellulose hydrogels crosslinked with different agents.

The maximum transient swelling strongly depended on the type of crosslinker. Citric-acid-crosslinked hydrogels reached peak swelling values of approximately 650–740 g/g, which can be attributed to their relatively low crosslinking density and high concentration of hydrophilic carboxyl groups. Epichlorohydrin-crosslinked hydrogels exhibited similarly high transient swelling (700–775 g/g), reflecting rapid water diffusion into a flexible but chemically crosslinked network. In contrast, MBAA-crosslinked hydrogels showed moderate peak swelling (600–670 g/g), while glutaraldehyde-crosslinked hydrogels exhibited the lowest transient swelling (500–570 g/g) due to their rigid acetal-linked network structure.

These results clearly indicate that decreasing crosslinking density and increasing hydrophilic functionality enhance the swelling performance of the hybrid hydrogels.

Figure 4 summarizes the true equilibrium swelling capacity of PAN–cellulose hydrogels measured after 24 h of immersion, when no further mass change was observed. In contrast to the transient maximum swelling shown in Figure 3, these values represent the stabilized network expansion under equilibrium conditions.

The comparative equilibrium swelling data summarized in Figure 4 further highlight the influence of crosslinker chemistry. The maximum equilibrium swelling values followed the order:

CA (≈540 g/g) > MBAA (≈510 g/g) >ECH (≈420 g/g) > GA (≈310 g/g).

4.2. Equilibrium Swelling Comparison

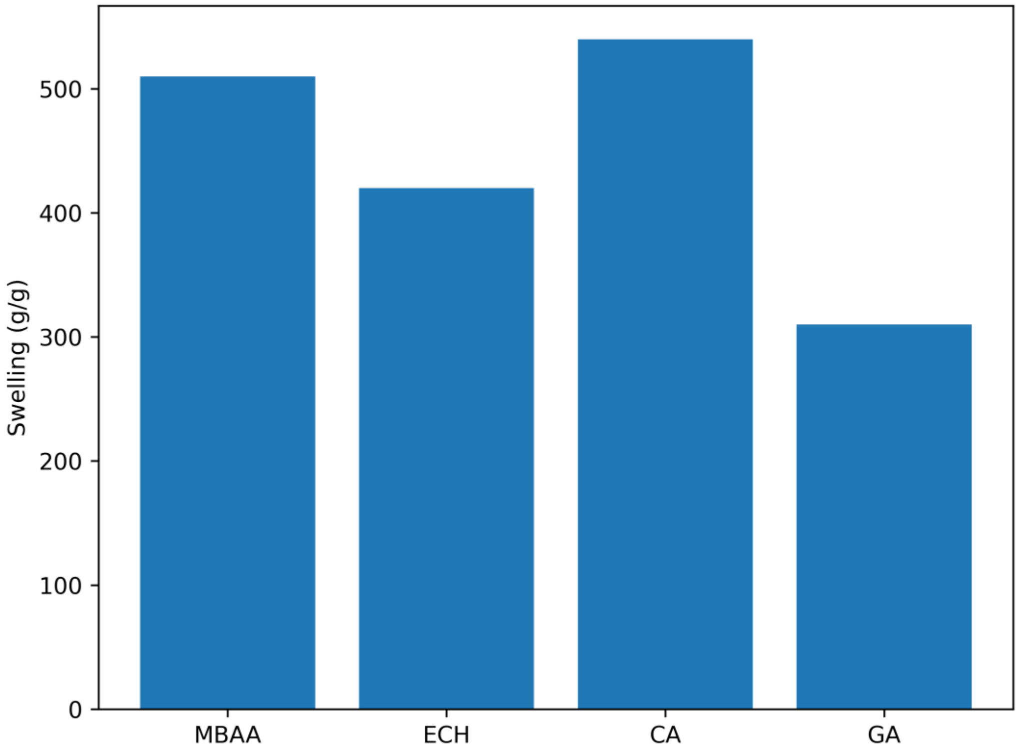


Figure 4. Equilibrium swelling capacity of PAN–cellulose hydrogels prepared with different crosslinkers.

Although epichlorohydrin-crosslinked hydrogels exhibited higher transient swelling during kinetic measurements (Figure 3), their equilibrium swelling was lower than that of citric-acid- and MBAA-crosslinked systems due to higher effective crosslinking density and restricted long-term chain relaxation.

This trend confirms that crosslinker selection is a key parameter for tailoring hydrogel swelling behavior for agricultural applications.

4.3. Soil Moisture Retention Performance

Figure 5 illustrates the soil moisture retention performance of hydrogel-amended soils over a 10-day period. Compared to the control soil, which lost moisture rapidly from **≈75% to ≈43%**, all hydrogel-treated soils exhibited significantly improved moisture retention.

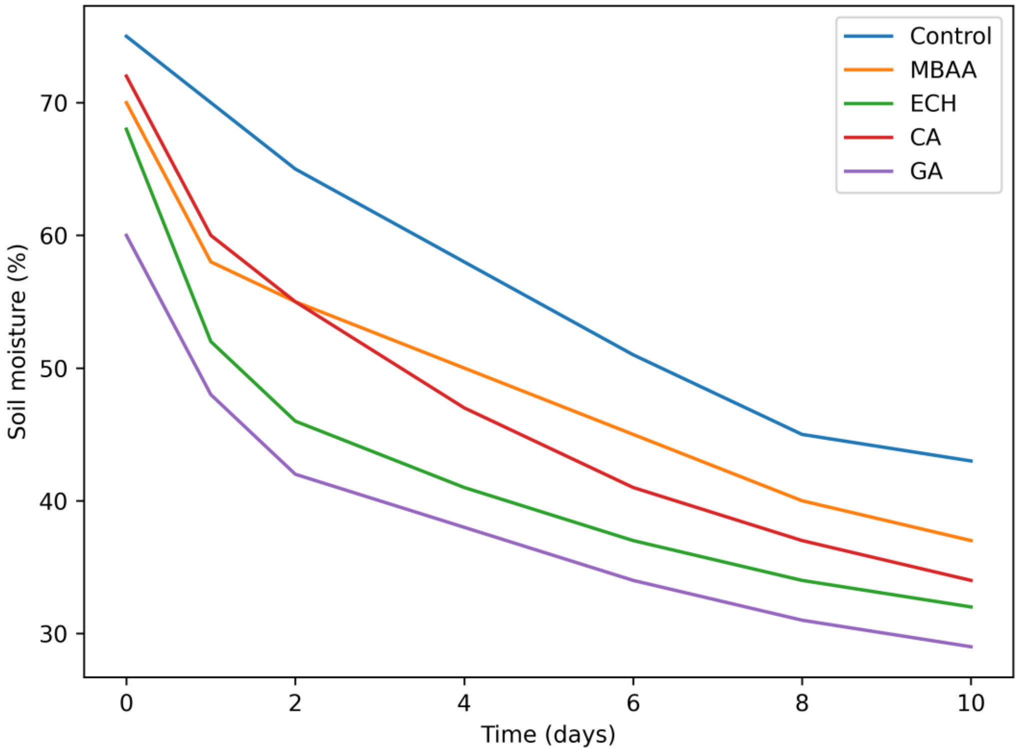


Figure 5. Soil moisture retention performance of hydrogel-amended soils compared to control soil.

Soils containing citric-acid-crosslinked hydrogels maintained water content above **≈55%** after 2 days and **≈34%** after 10 days, demonstrating superior water-holding capacity. Epichlorohydrin-based hydrogels showed comparable performance, retaining **≈52%** moisture at day 1 and **≈32%** at day 10. MBAA-crosslinked hydrogels exhibited moderate retention (**≈37% at day 10**), whereas glutaraldehyde-based hydrogels showed the least improvement due to their lower swelling capacity. Overall, the use of CA- and ECH-crosslinked hydrogels resulted in an estimated **25–30% reduction in irrigation demand**, highlighting their strong potential for water-saving agricultural practices.

4.4. Biodegradability under Soil Burial Conditions

The biodegradability of PAN–cellulose hybrid hydrogels was evaluated by monitoring their weight loss under soil burial conditions over a period of three months (Figure 6). All samples exhibited a gradual increase in weight loss with increasing burial time, indicating progressive degradation of the hydrogel systems in the soil environment.

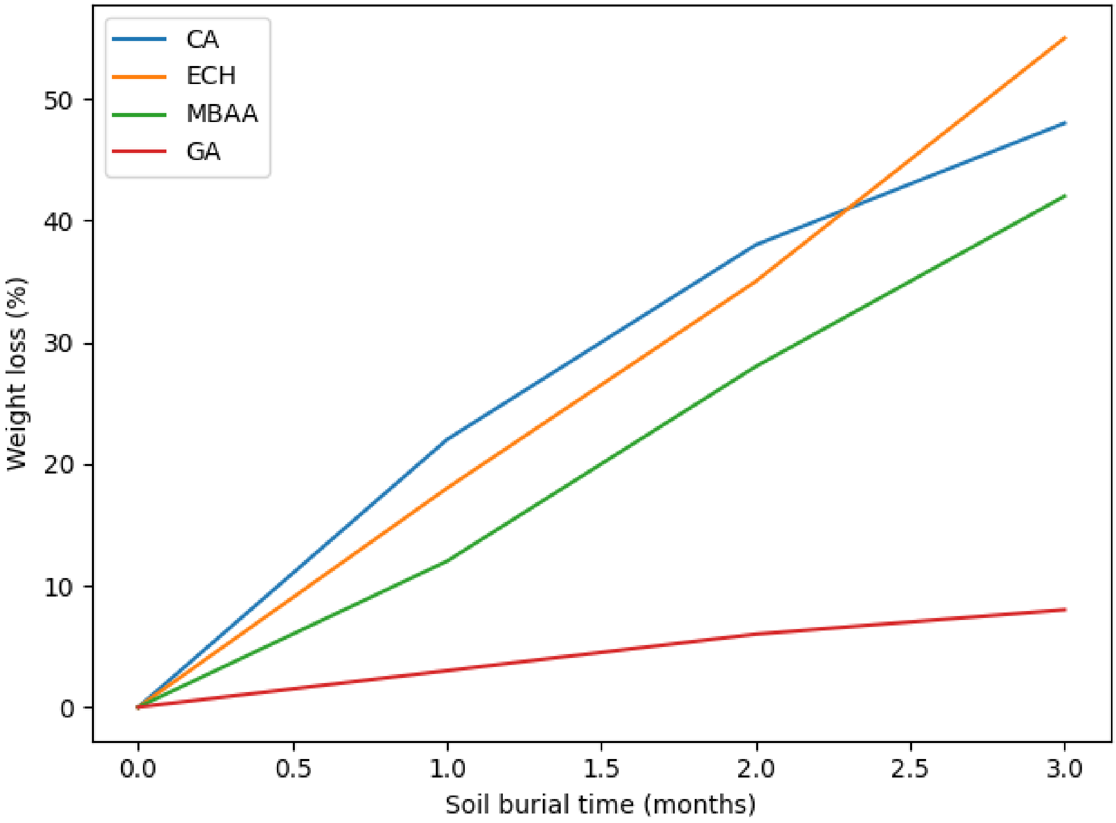


Figure 6. Weight loss behavior of PAN–cellulose hydrogels crosslinked with different agents under soil burial conditions over three months.

Citric-acid-crosslinked hydrogels showed pronounced biodegradation, with weight loss increasing from approximately 22% after the first month to about 48% after three months. This behavior can be attributed to the presence of ester linkages and a relatively loose network structure, which are susceptible to hydrolytic and microbial degradation.

Epichlorohydrin-crosslinked hydrogels exhibited moderate-to-high biodegradation, reaching approximately 55% weight loss after three months of soil burial. The enhanced degradation compared to MBAA-crosslinked systems is associated with the hydrolysis of ether linkages and gradual disintegration of the PAN–cellulose network.

MBAA-crosslinked hydrogels demonstrated moderate biodegradability, with weight loss values of approximately 12%, 28%, and 42% after one, two, and three months, respectively. This weight loss is primarily attributed to the biodegradation of the cellulose component and the leaching of low-molecular-weight hydrolyzed PAN fragments, while the covalently crosslinked MBAA network itself remained relatively stable.

In contrast, glutaraldehyde-crosslinked hydrogels exhibited minimal biodegradation, with weight loss not exceeding 8% even after three months. The rigid acetal-linked network structure effectively restricted water penetration and microbial attack, resulting in high structural stability under soil burial conditions.

4.5. Overall Performance Assessment and Practical Implications

By correlating swelling behavior, soil moisture retention, and biodegradability, it becomes evident that citric acid and epichlorohydrin provide the most favorable balance between performance and environmental compatibility.

Although epichlorohydrin is classified as a potentially hazardous compound, its role in the present study is limited to the crosslinking stage, and extensive post-synthesis washing ensures the removal of unreacted residues. As a result, the final hydrogel materials contain covalently bound ether linkages and pose minimal environmental risk when applied to soil under agricultural conditions.

Citric acid is particularly suitable for short-term agricultural applications requiring high water absorption and rapid biodegradation, while epichlorohydrin offers a compromise between mechanical stability and biodegradability. These findings demonstrate that hybrid hydrogels derived from PAN carpet waste and cotton linter can be effectively tailored through crosslinker selection, enabling their optimization for sustainable agriculture in water-limited regions.

5. Conclusions

In this work, hybrid biodegradable hydrogels were successfully synthesized from polyacrylonitrile (PAN)-based carpet manufacturing waste and cotton linter through alkaline hydrolysis followed by crosslinking with different agents. The conversion of nitrile (–CN) groups in PAN into hydrophilic carboxylate ($-COO^-$) and amide ($-CONH_2$) functionalities, combined with hydroxyl-rich cellulose chains from cotton linter, enabled the formation of three-dimensional PAN–cellulose hydrogel networks with tunable properties.

The results demonstrated that the type of crosslinking agent plays a decisive role in controlling hydrogel performance. Citric-acid- and epichlorohydrin-crosslinked hydrogels exhibited the highest transient (up to ≈740 g/g) and equilibrium swelling capacities (≈520–560 g/g), along with superior soil moisture retention and favorable biodegradability. In contrast, MBAA-crosslinked hydrogels showed moderate swelling and degradation behavior, while glutaraldehyde-crosslinked hydrogels exhibited limited swelling **(**≈310 g/g**)** and minimal biodegradability (≈8% weight loss after three months) due to their rigid network structure.

Soil moisture retention studies revealed that hydrogels crosslinked with citric acid and epichlorohydrin reduced water loss significantly compared to untreated soil, leading to an estimated 25–30% reduction in irrigation demand**.** These findings highlight the strong potential of PAN–cellulose hybrid hydrogels as water-retaining soil amendments for sustainable agriculture, particularly in arid and semi-arid regions.

Overall, this study demonstrates a cost-effective and environmentally friendly waste-to-resource strategy that simultaneously addresses textile waste management and agricultural water scarcity. The developed hybrid hydrogels, especially those crosslinked with bio-based citric acid, represent a promising class of biodegradable materials for sustainable soil moisture management. Future work will focus on long-term field trials, mechanical stability under agricultural conditions, and large-scale processing feasibility.

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